Chemical and Physical Properties of Hydrogen Peroxide

Density - 1.46 g/ml
Boiling Point - 152 C
Critical Temperature - 457 C
Critical Pressure - 214 atm
Molar Enthalpy of Vaporization - 51 kJ/mole
Solubility Parameter - 46 $MPa^{1/2}$

FIG. 1

Chemical and Physical Properties of Carbon Dioxide

Density - 0.4 -0.9 g/ml
Critical Temperature - 31 C
Critical Pressure - 73 atm
Molar Enthalpy of Vaporization - 2.5 kJ/mole
Solubility Parameter - 14-22 $MPa^{1/2}$

CO_2 Permeability with HDPE Film

He	CO_2
0.005	2
360	16
1.9	[31]

16x more permeable than He

Permeability = Diffusivity x Solubility

CO_2 compared to EtO

	EtO	CO_2
S	22	22
MW	44	44

Solubility Parameter - $MPa^{1/2}$

Molecular Weight g/mole

Sporicidal Activity

EtO epoxide group
CO_2 oxy acid group (H_2O_2)

FIG. 2

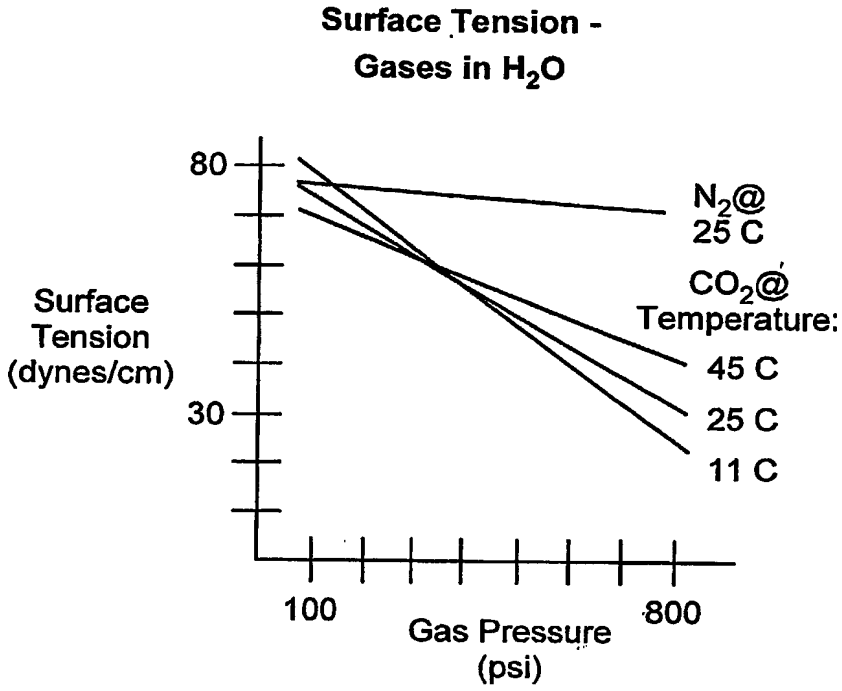

Surface Tension -
Gases in H_2O
80
30
Surface
Tension
(dynes/cm)
N_2@
25 C
CO_2@
Temperature:
45 C
25 C
11 C
100
800
Gas Pressure
(psi)

FIG. 3

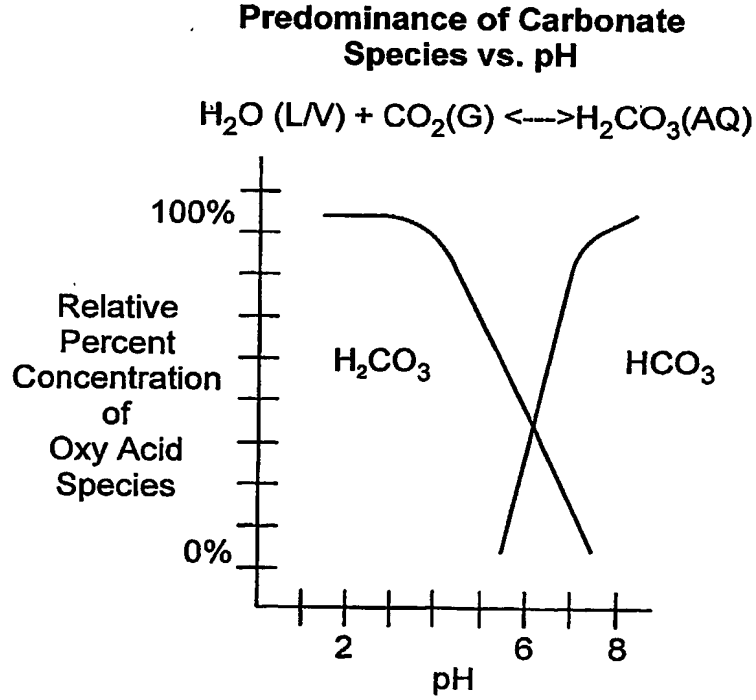

Predominance of Carbonate
Species vs. pH
H_2O (L/V) + CO_2(G) <---> H_2CO_3(AQ)
100%
0%
Relative
Percent
Concentration
of
Oxy Acid
Species
H_2CO_3
HCO_3
2
6
8
pH

FIG. 4

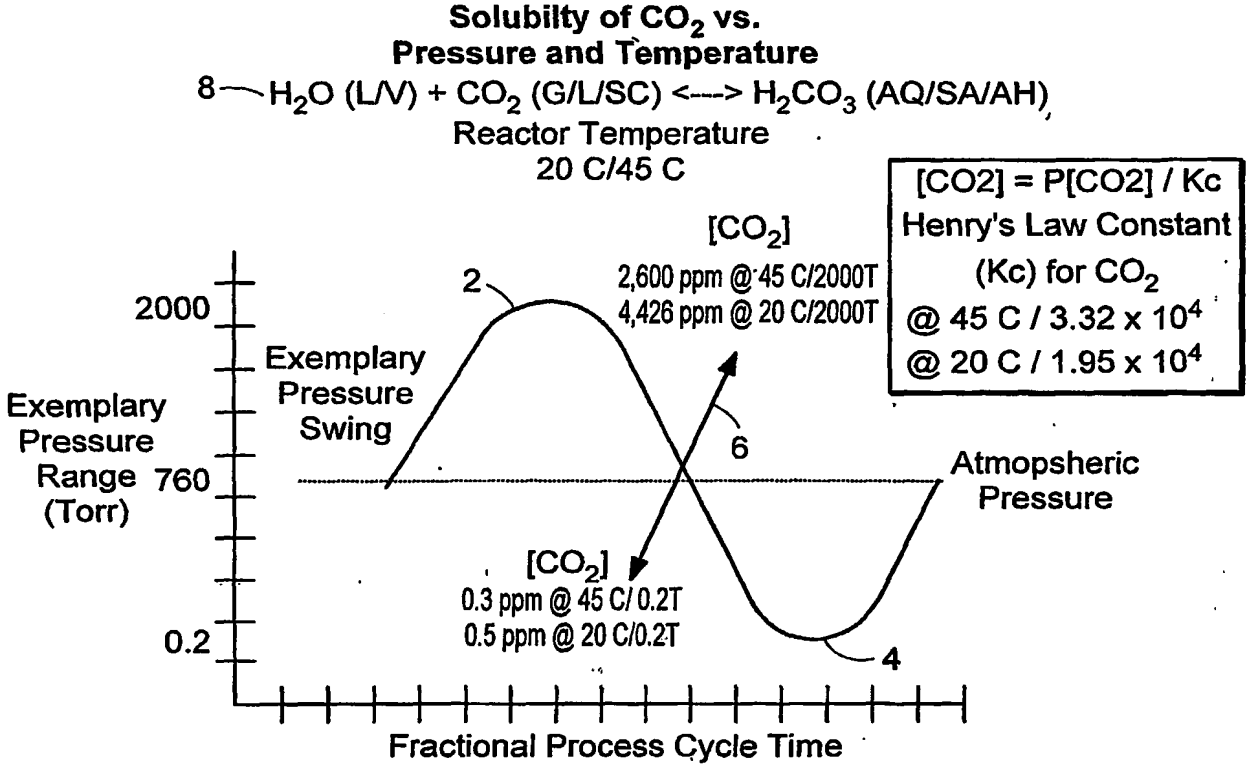

Solubilty of CO_2 vs.
Pressure and Temperature
8 H_2O (L/V) + CO_2 (G/L/SC) <---> H_2CO_3 (AQ/SA/AH)
Reactor Temperature
20 C/45 C
[CO2] = P[CO2] / Kc
Henry's Law Constant
(Kc) for CO_2
@ 45 C / 3.32 x 10^4
@ 20 C / 1.95 x 10^4
$[CO_2]$
2,600 ppm @ 45 C/2000T
4,426 ppm @ 20 C/2000T
2
2000
Exemplary
Pressure
Swing
Exemplary
Pressure
Range
(Torr)
760
6
Atmopsheric
Pressure
$[CO_2]$
0.3 ppm @ 45 C/ 0.2T
0.5 ppm @ 20 C/0.2T
0.2
4
Fractional Process Cycle Time

FIG. 5

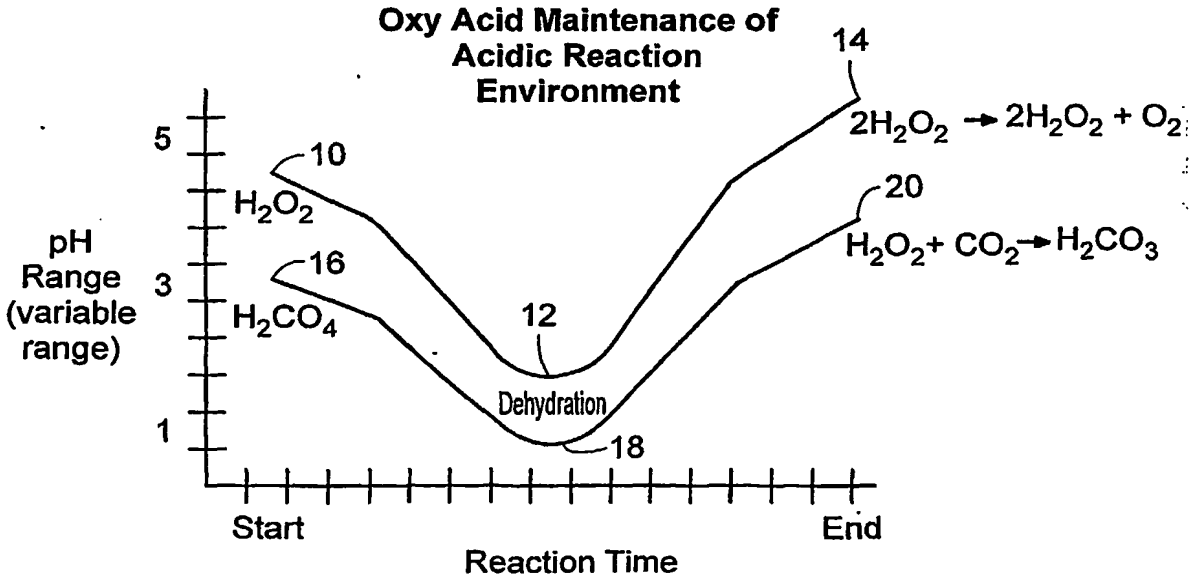

Oxy Acid Maintenance of
Acidic Reaction
Environment
14
$2H_2O_2 \rightarrow 2H_2O + O_2$
5
10
H_2O_2
20
$H_2O_2 + CO_2 \rightarrow H_2CO_3$
pH
Range
(variable
range)
3
16
H_2CO_4
12
Dehydration
1
18
Start
End
Reaction Time

FIG. 6

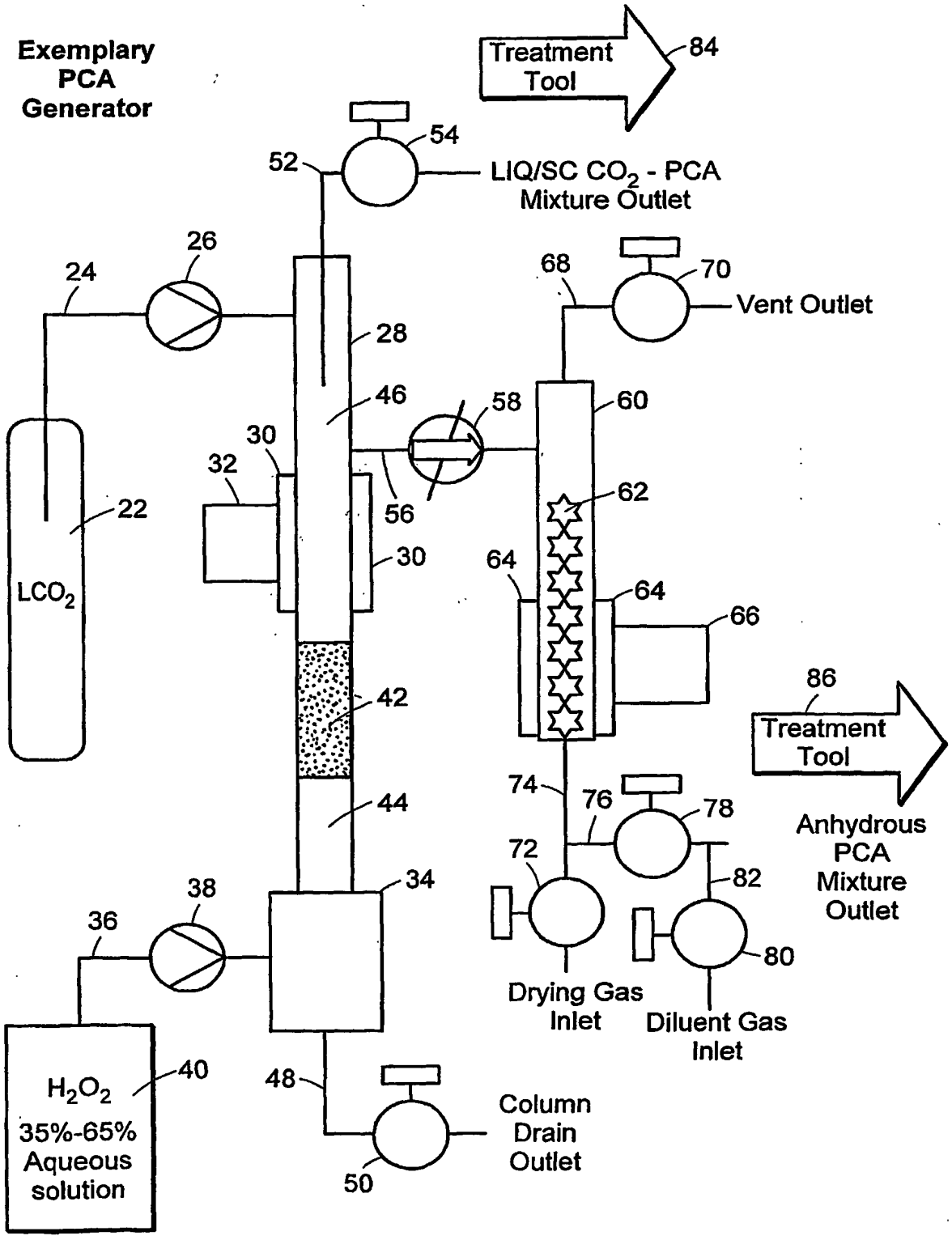
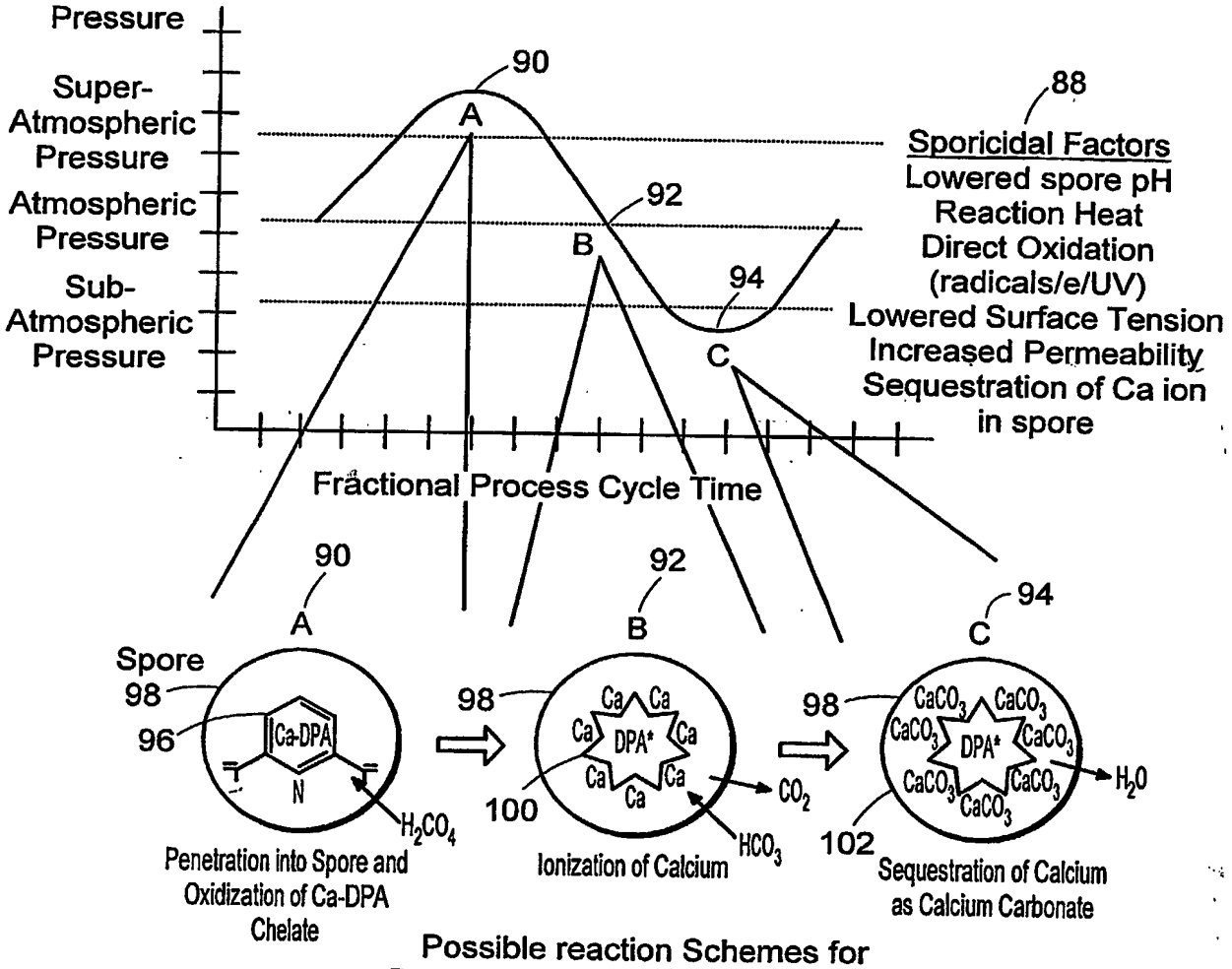

Exemplary PCA Generator
Treatment Tool
84
54
52
LIQ/SC CO_2 - PCA Mixture Outlet
26
24
68
70
Vent Outlet
28
46
58
60
30
32
56
62
22
30
64
64
66
LCO_2
42
86
Treatment Tool
74
76
78
Anhydrous PCA Mixture Outlet
44
72
82
38
34
36
80
Drying Gas Inlet
Diluent Gas Inlet
40
48
H_2O_2
35%-65% Aqueous solution
Column Drain Outlet
50

FIG. 7

Possible Mechanisms for Sporicidal Action of Percarbonic Acid Treatment



Possible reaction Schemes for Percarbonate (PCA) Oxidation and Sequestration of Spore Complex Ca-DPA

104 Eq1: $CO_2 + H_2O_2 <---> H_2CO_4$

106 Eq2: $H_2CO_4 + \text{Ca-DPA} <---> Ca^{2+} + \text{DPA*} + CO_2 + H_2O$

108 Eq3: $CO_2 + H_2O <---> H + HCO_3$

110 Eq4: $Ca^{2+} + 2HCO_3 <---> CaCO_3 + CO_2 + H_2O$

DPA* -Oxidized DPA structure

FIG. 8

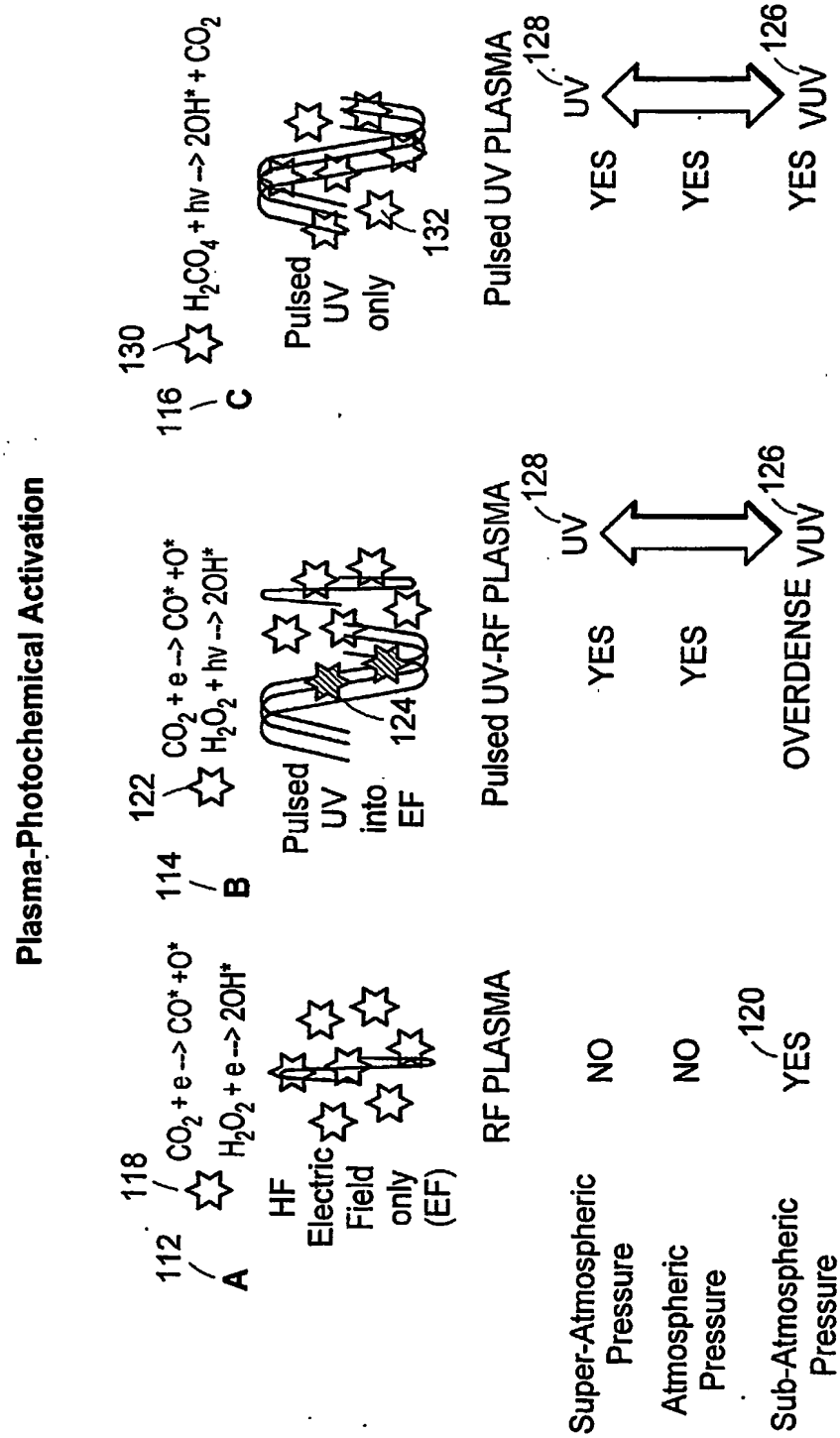

Plasma-Photochemical Activation
112
A
118
$CO_2 + e \rightarrow CO^* + O^*$
$H_2O_2 + e \rightarrow 2OH^*$
HF Electric Field only (EF)
114
B
122
$CO_2 + e \rightarrow CO^* + O^*$
$H_2O_2 + h\nu \rightarrow 2OH^*$
Pulsed UV into EF
124
116
C
130
$H_2CO_4 + h\nu \rightarrow 2OH^* + CO_2$
Pulsed UV only
132
RF PLASMA
Pulsed UV-RF PLASMA
Pulsed UV PLASMA
Super-Atmospheric Pressure
NO
YES
YES
Atmospheric Pressure
NO
YES
YES
Sub-Atmospheric Pressure
120
YES
OVERDENSE
YES
128
UV
126
VUV

FIG. 9

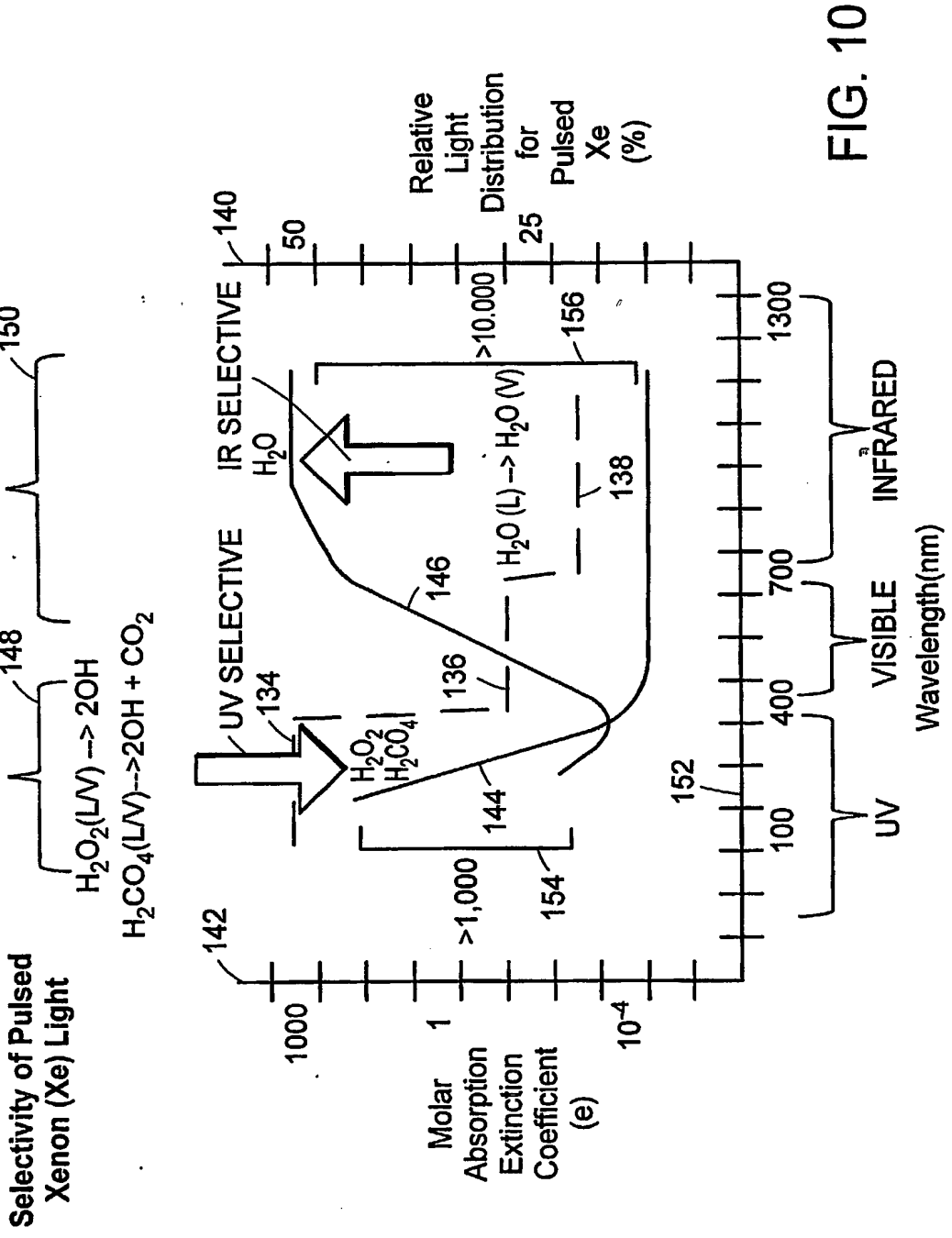

Selectivity of Pulsed Xenon (Xe) Light
RADICALIZING EFFECT
148
H_2O_2(L/V) --> 2OH
H_2CO_4(L/V)-->2OH + CO_2
DEHYDRATION EFFECT
150
UV SELECTIVE
134
IR SELECTIVE
H_2O
140
H_2O_2
H_2CO_4
136
H_2O (L) --> H_2O (V)
138
146
144
154
152
156
142
1000
>1,000
1
10^{-4}
>10.000
50
25
Molar Absorption Extinction Coefficient (e)
Relative Light Distribution for Pulsed Xe (%)
100
400
700
1300
UV
VISIBLE
INFRARED
Wavelength(nm)

FIG. 10

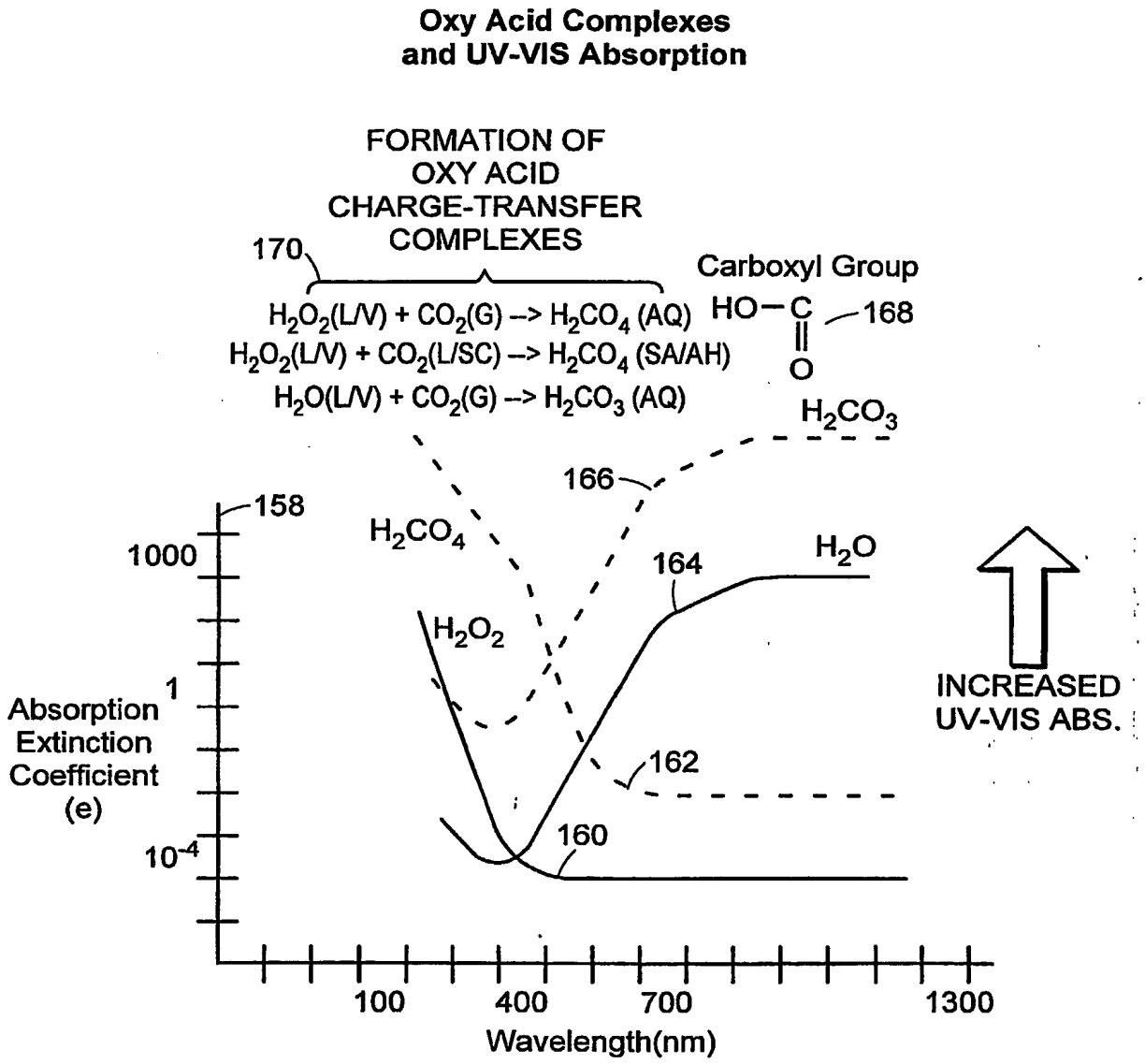

Oxy Acid Complexes
and UV-VIS Absorption
FORMATION OF
OXY ACID
CHARGE-TRANSFER
COMPLEXES
170
$H_2O_2(L/V) + CO_2(G) \rightarrow H_2CO_4 (AQ)$
$H_2O_2(L/V) + CO_2(L/SC) \rightarrow H_2CO_4 (SA/AH)$
$H_2O(L/V) + CO_2(G) \rightarrow H_2CO_3 (AQ)$
Carboxyl Group
HO—C
O
168
H_2CO_3
166
158
H_2CO_4
1000
164
H_2O
H_2O_2
1
Absorption
Extinction
Coefficient
(e)
10^{-4}
INCREASED
UV-VIS ABS.
162
160
100
400
700
1300
Wavelength(nm)

FIG. 11

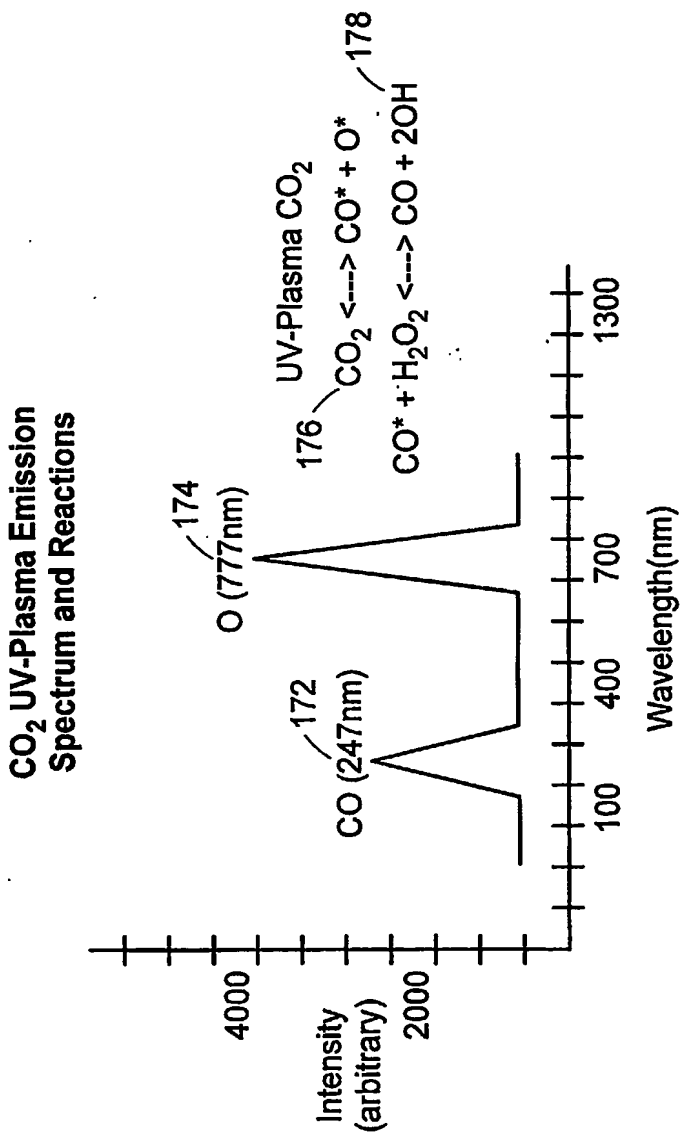

CO_2 UV-Plasma Emission
Spectrum and Reactions
174
O (777nm)
172
CO (247nm)
176
UV-Plasma CO_2
CO_2 <---> CO* + O*
178
CO* + H_2O_2 <---> CO + 2OH
Intensity (arbitrary)
4000
2000
100
400
700
1300
Wavelength(nm)

FIG. 12

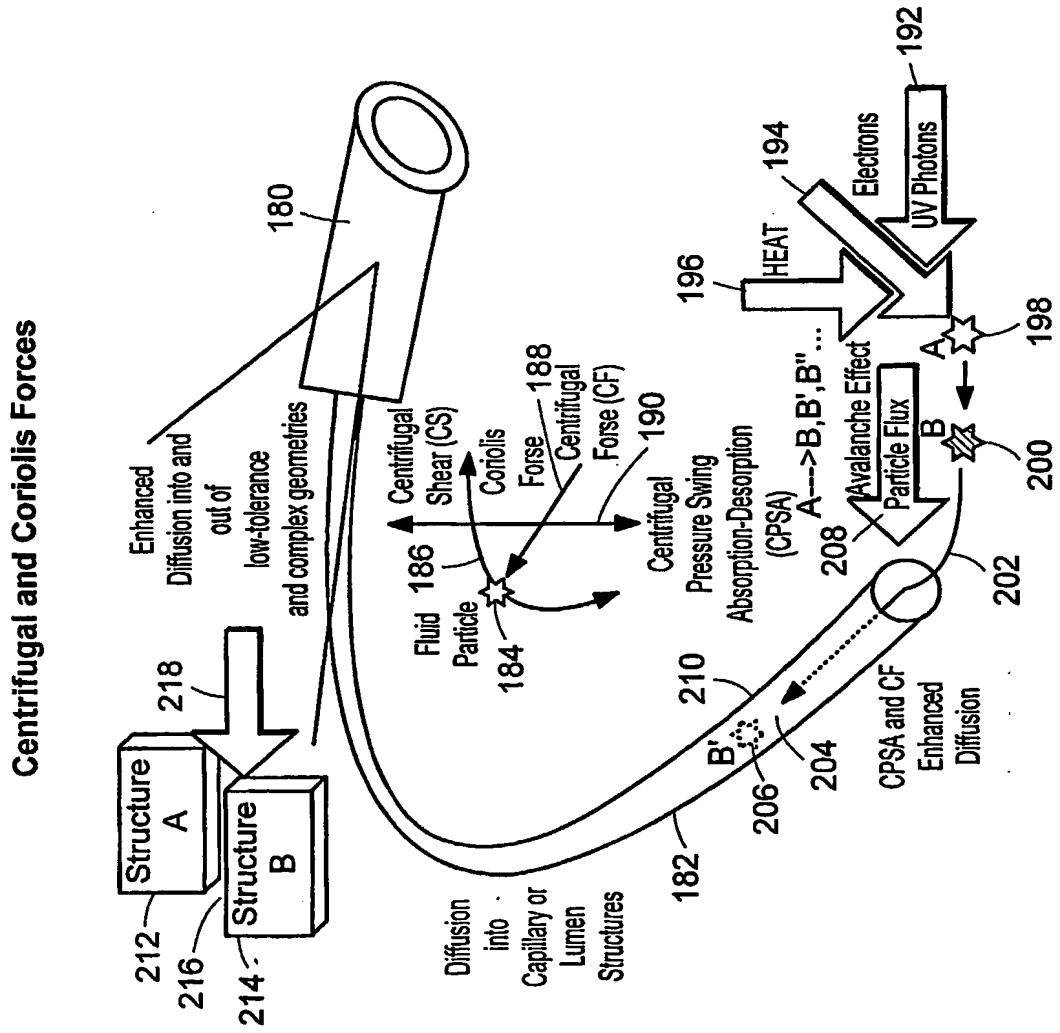

Centrifugal and Coriolis Forces
Structure A
Structure B
212
216
214
218
Enhanced Diffusion into and out of low-tolerance and complex geometries
180
Diffusion into Capillary or Lumen Structures
182
Fluid Particle
184
186
Centrifugal Shear (CS)
Coriolis Forse
Centrifugal Forse (CF)
188
190
Centrifugal Pressure Swing Absorption-Desorption (CPSA)
A-->B,B',B"...
208
B'
210
206
204
CPSA and CF Enhanced Diffusion
202
Avalanche Effect
Particle Flux
B
200
A
198
HEAT
196
Electrons
UV Photons
194
192

FIG. 13

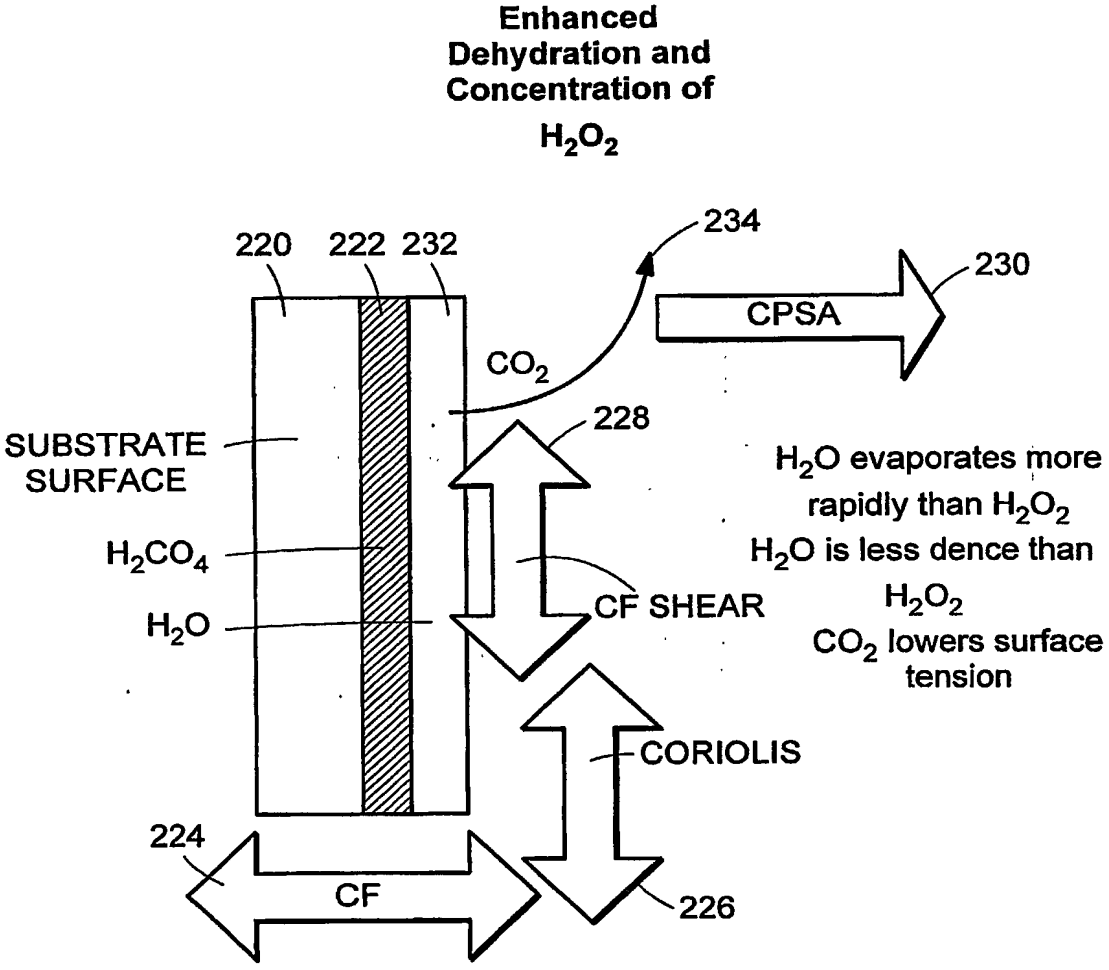

Enhanced
Dehydration and
Concentration of
H_2O_2
220
222
232
234
230
CPSA
CO_2
228
SUBSTRATE
SURFACE
H_2CO_4
H_2O
CF SHEAR
CORIOLIS
224
CF
226
H_2O evaporates more
rapidly than H_2O_2
H_2O is less dence than
H_2O_2
CO_2 lowers surface
tension

FIG. 14

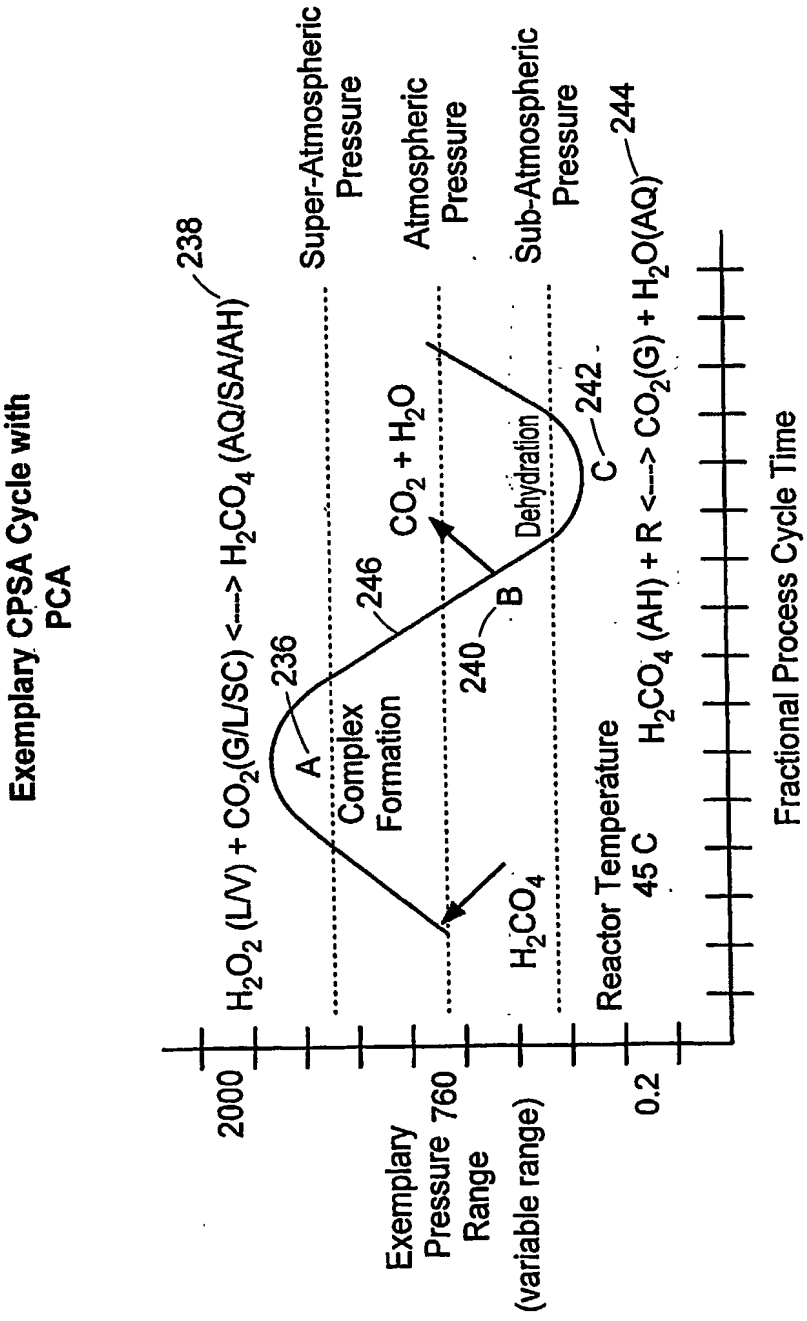

Exemplary CPSA Cycle with PCA
H_2O_2 (L/V) + CO_2(G/L/SC) <---> H_2CO_4 (AQ/SA/AH)
238
Super-Atmospheric Pressure
Atmospheric Pressure
Sub-Atmospheric Pressure
A
236
Complex Formation
246
CO_2 + H_2O
B
240
Dehydration
C
242
H_2CO_4
Reactor Temperature
45 C
H_2CO_4 (AH) + R <---> CO_2(G) + H_2O(AQ)
244
2000
Exemplary Pressure Range (variable range)
760
0.2
Fractional Process Cycle Time

FIG. 15

Exemplary Centrifugal UV-Plasma Treatment Apparatus (Vertical Basket)

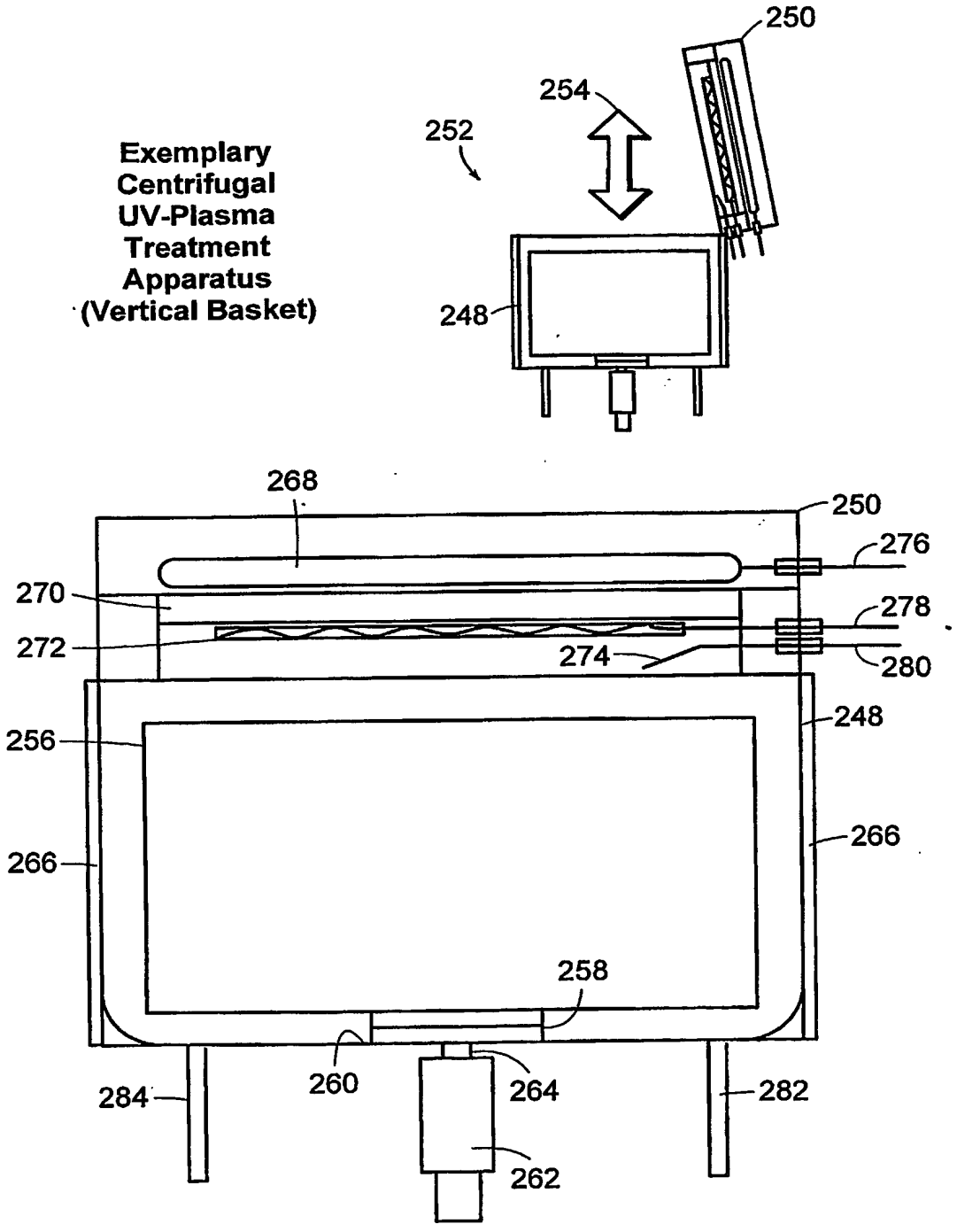


FIG. 16

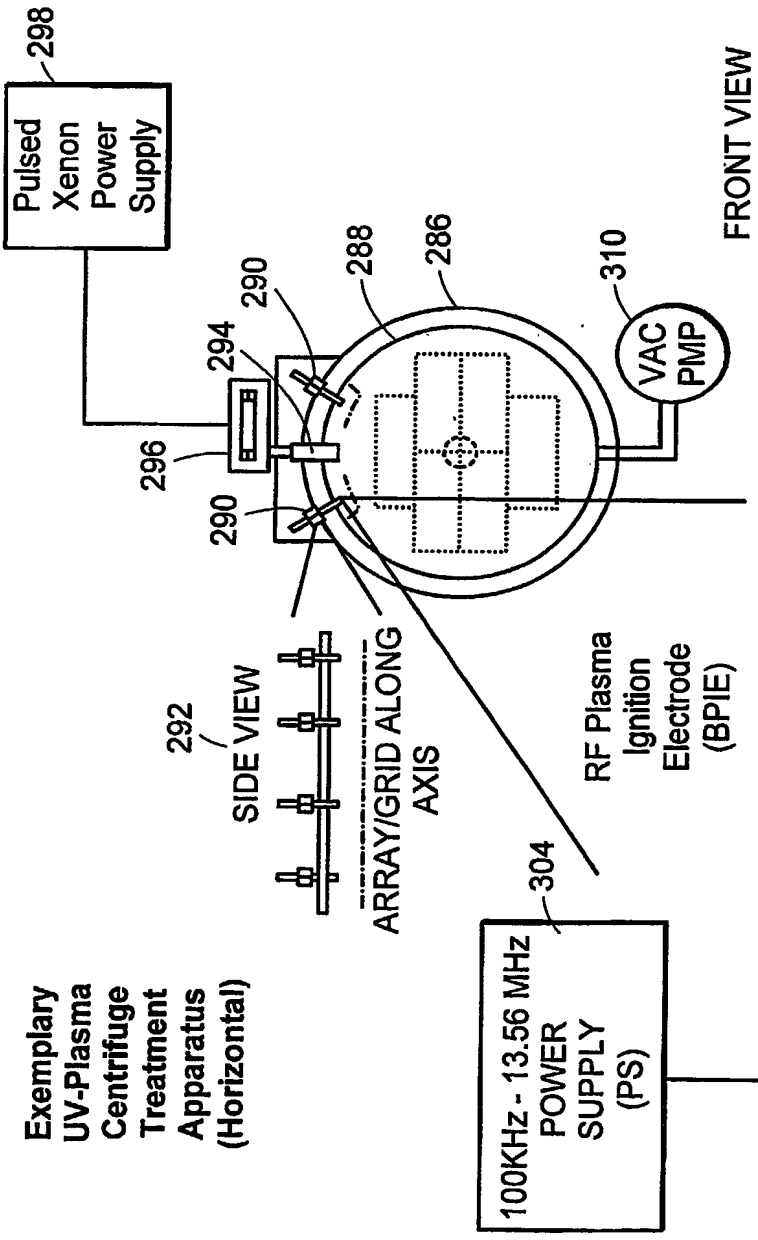
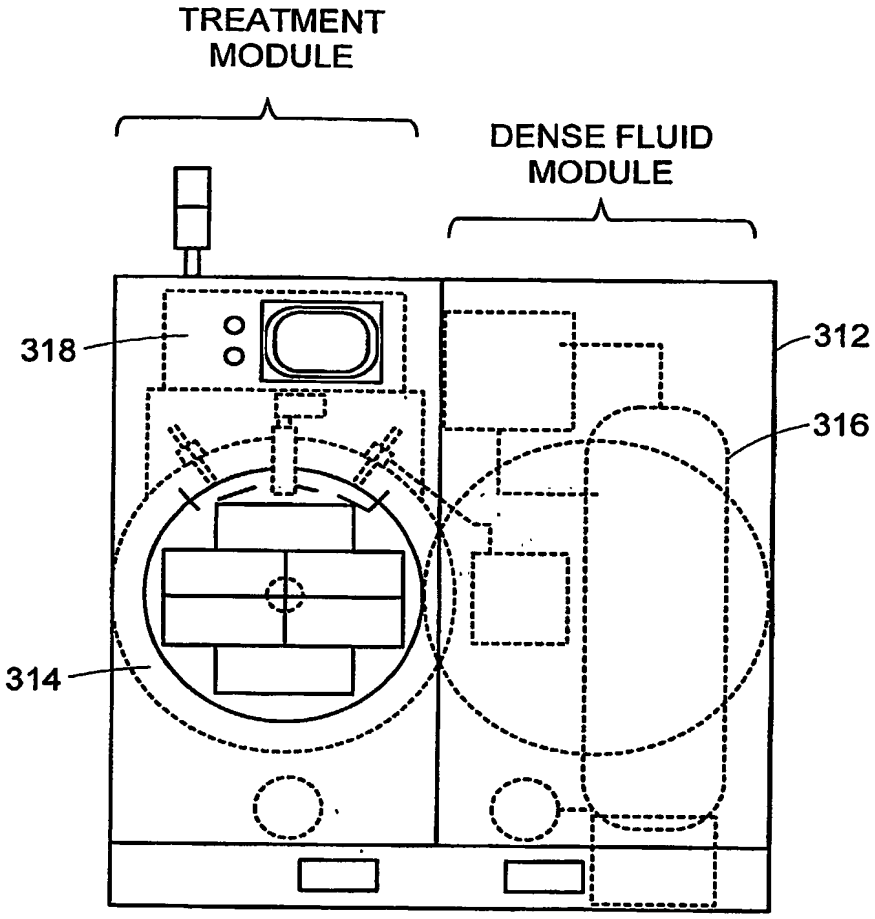

Exemplary UV-Plasma Centrifuge Treatment Apparatus (Horizontal)
Pulsed Xenon Power Supply
298
296
294
290
288
286
310
VAC PMP
FRONT VIEW
INTERIOR
308
306
300
302
304
100KHz - 13.56 MHz POWER SUPPLY (PS)
RF Plasma Ignition Electrode (BPIE)
292
SIDE VIEW
ARRAY/GRID ALONG AXIS

FIG. 17



Exemplary Centifugal UV-Plasma Treatment System Design

FIG. 18

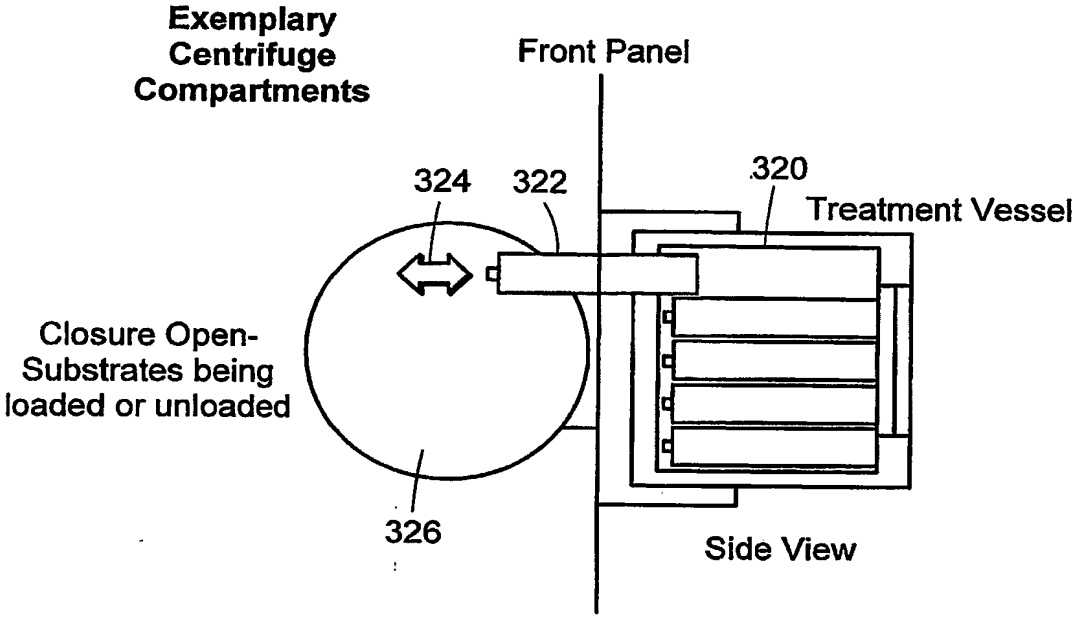


FIG. 19A

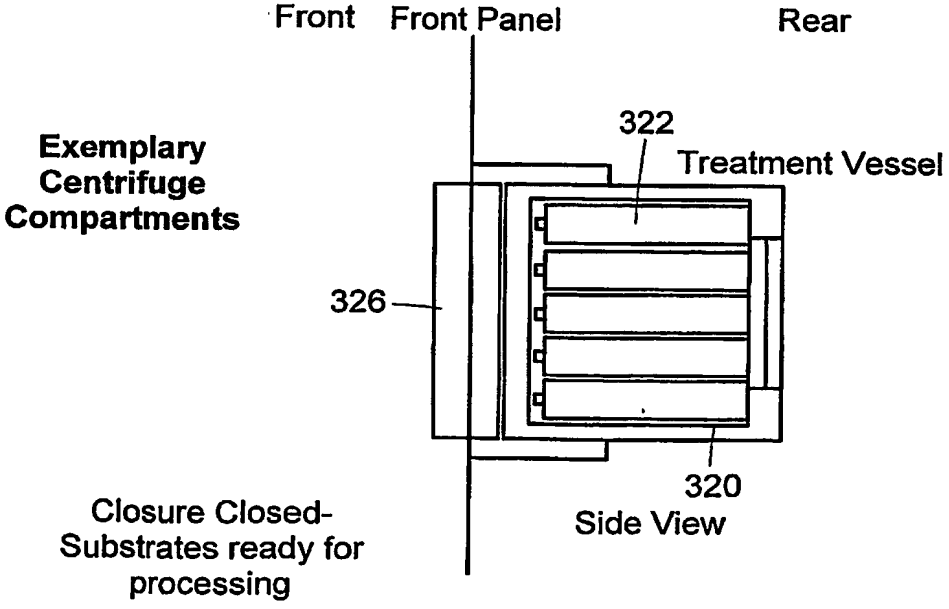


FIG. 19B

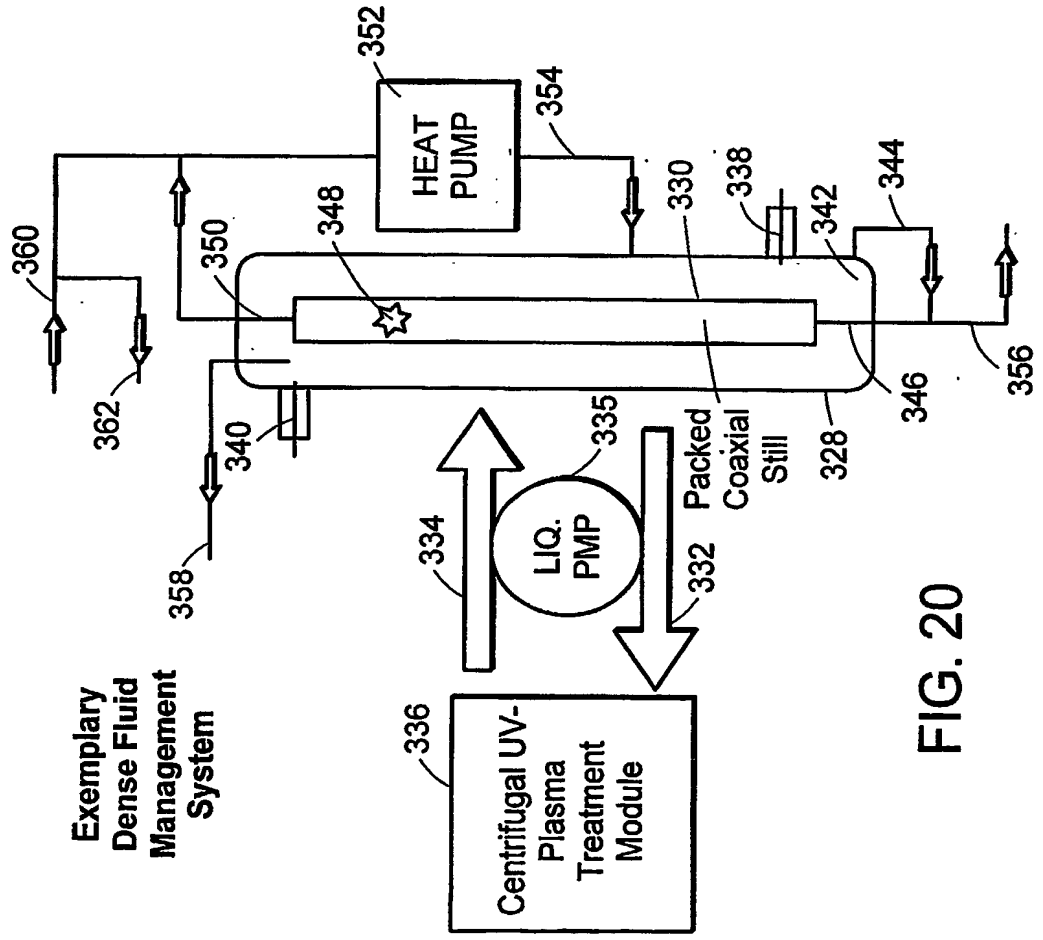

352
HEAT PUMP
354
330
338
342
344
348
350
360
362
340
346
356
328
Packed Coaxial Still
335
LIQ. PMP
334
332
358
336
Centrifugal UV-Plasma Treatment Module
Exemplary Dense Fluid Management System

FIG. 20

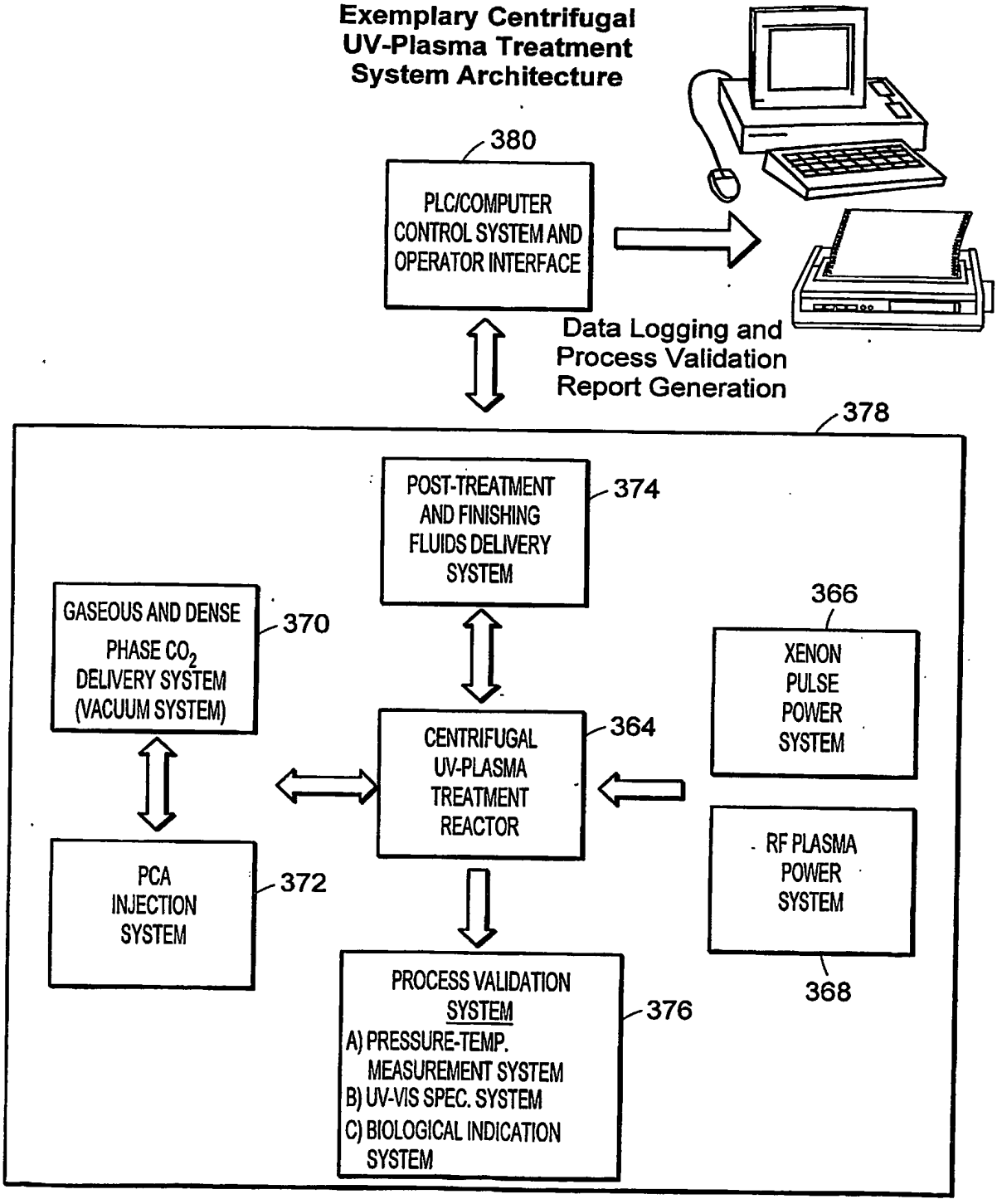

Exemplary Centrifugal UV-Plasma Treatment System Architecture
380
PLC/COMPUTER CONTROL SYSTEM AND OPERATOR INTERFACE
Data Logging and Process Validation Report Generation
378
POST-TREATMENT AND FINISHING FLUIDS DELIVERY SYSTEM
374
GASEOUS AND DENSE PHASE CO_2 DELIVERY SYSTEM (VACUUM SYSTEM)
370
366
XENON PULSE POWER SYSTEM
CENTRIFUGAL UV-PLASMA TREATMENT REACTOR
364
PCA INJECTION SYSTEM
372
RF PLASMA POWER SYSTEM
368
PROCESS VALIDATION SYSTEM
A) PRESSURE-TEMP. MEASUREMENT SYSTEM
B) UV-VIS SPEC. SYSTEM
C) BIOLOGICAL INDICATION SYSTEM
376

FIG. 21

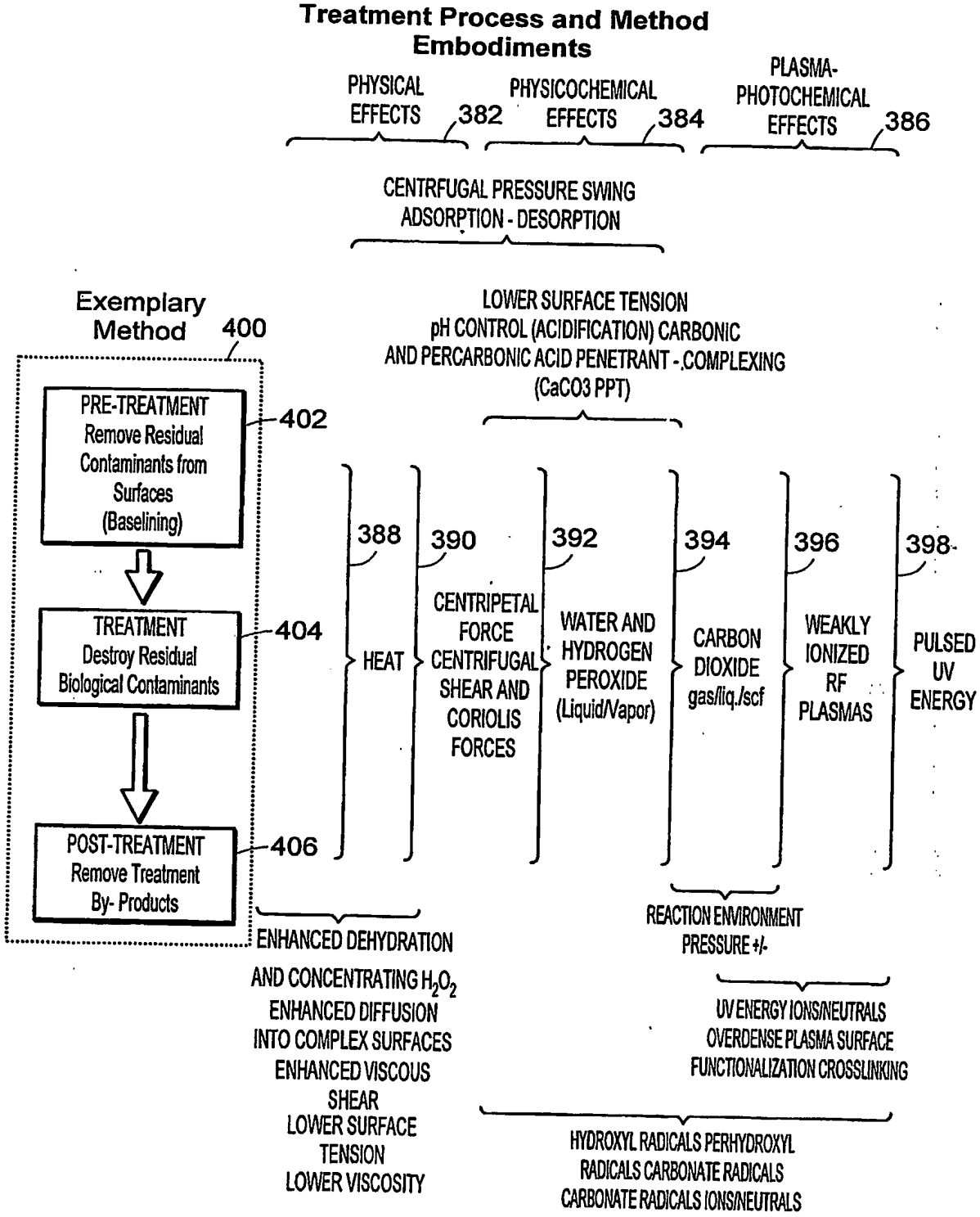
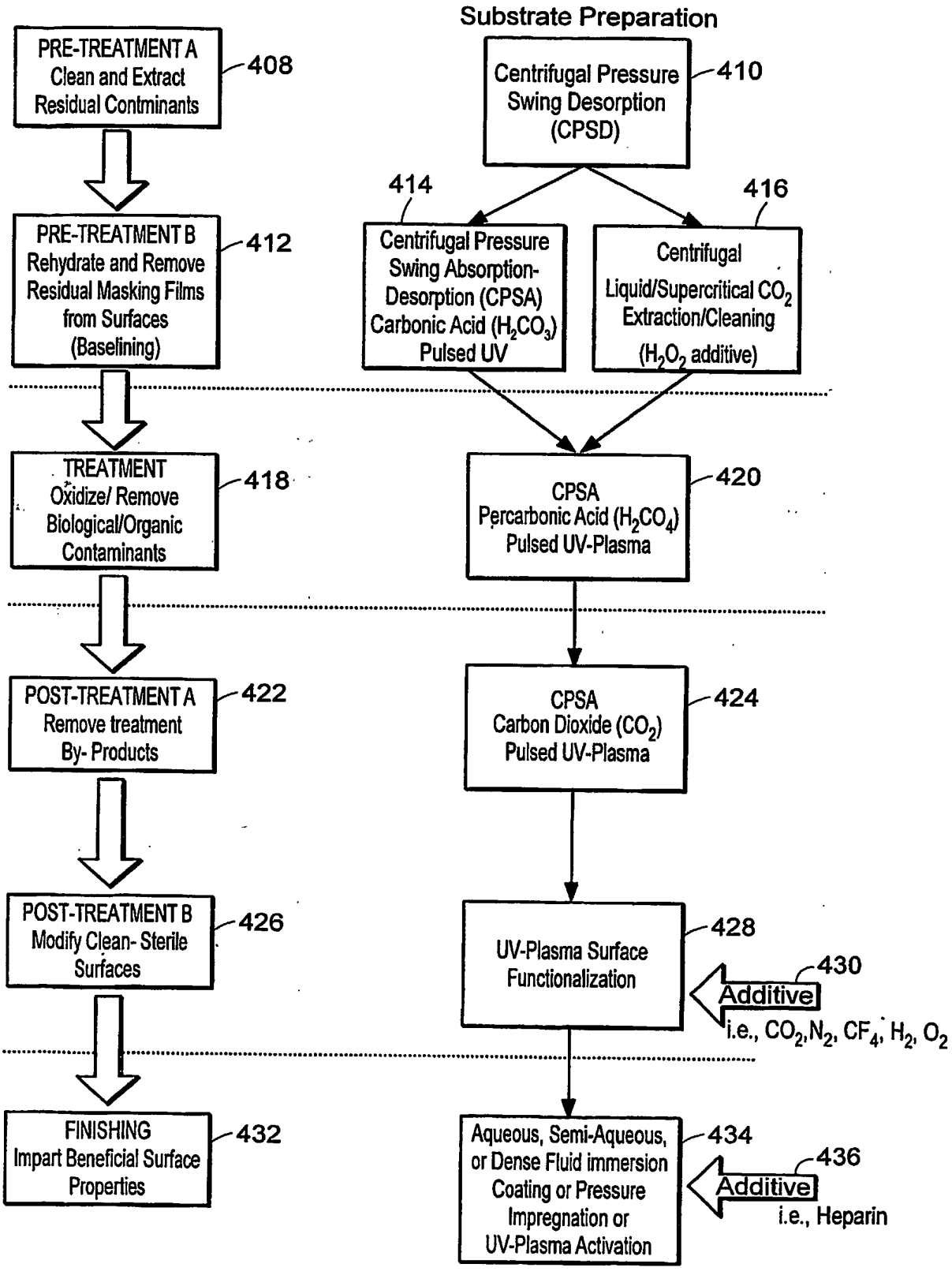

Treatment Process and Method Embodiments
PHYSICAL EFFECTS
382
PHYSICOCHEMICAL EFFECTS
384
PLASMA-PHOTOCHEMICAL EFFECTS
386
CENTRFUGAL PRESSURE SWING ADSORPTION - DESORPTION
Exemplary Method
400
LOWER SURFACE TENSION
pH CONTROL (ACIDIFICATION) CARBONIC AND PERCARBONIC ACID PENETRANT - COMPLEXING (CaCO3 PPT)
PRE-TREATMENT
Remove Residual Contaminants from Surfaces (Baselining)
402
TREATMENT
Destroy Residual Biological Contaminants
404
POST-TREATMENT
Remove Treatment By- Products
406
388
HEAT
390
CENTRIPETAL FORCE CENTRIFUGAL SHEAR AND CORIOLIS FORCES
392
WATER AND HYDROGEN PEROXIDE (Liquid/Vapor)
394
CARBON DIOXIDE gas/liq./scf
396
WEAKLY IONIZED RF PLASMAS
398
PULSED UV ENERGY
ENHANCED DEHYDRATION
AND CONCENTRATING H_2O_2
ENHANCED DIFFUSION INTO COMPLEX SURFACES
ENHANCED VISCOUS SHEAR
LOWER SURFACE TENSION
LOWER VISCOSITY
REACTION ENVIRONMENT PRESSURE +/-
UV ENERGY IONS/NEUTRALS
OVERDENSE PLASMA SURFACE FUNCTIONALIZATION CROSSLINKING
HYDROXYL RADICALS PERHYDROXYL RADICALS CARBONATE RADICALS
CARBONATE RADICALS IONS/NEUTRALS

FIG. 22

FIG. 23

Process and Method Architecture



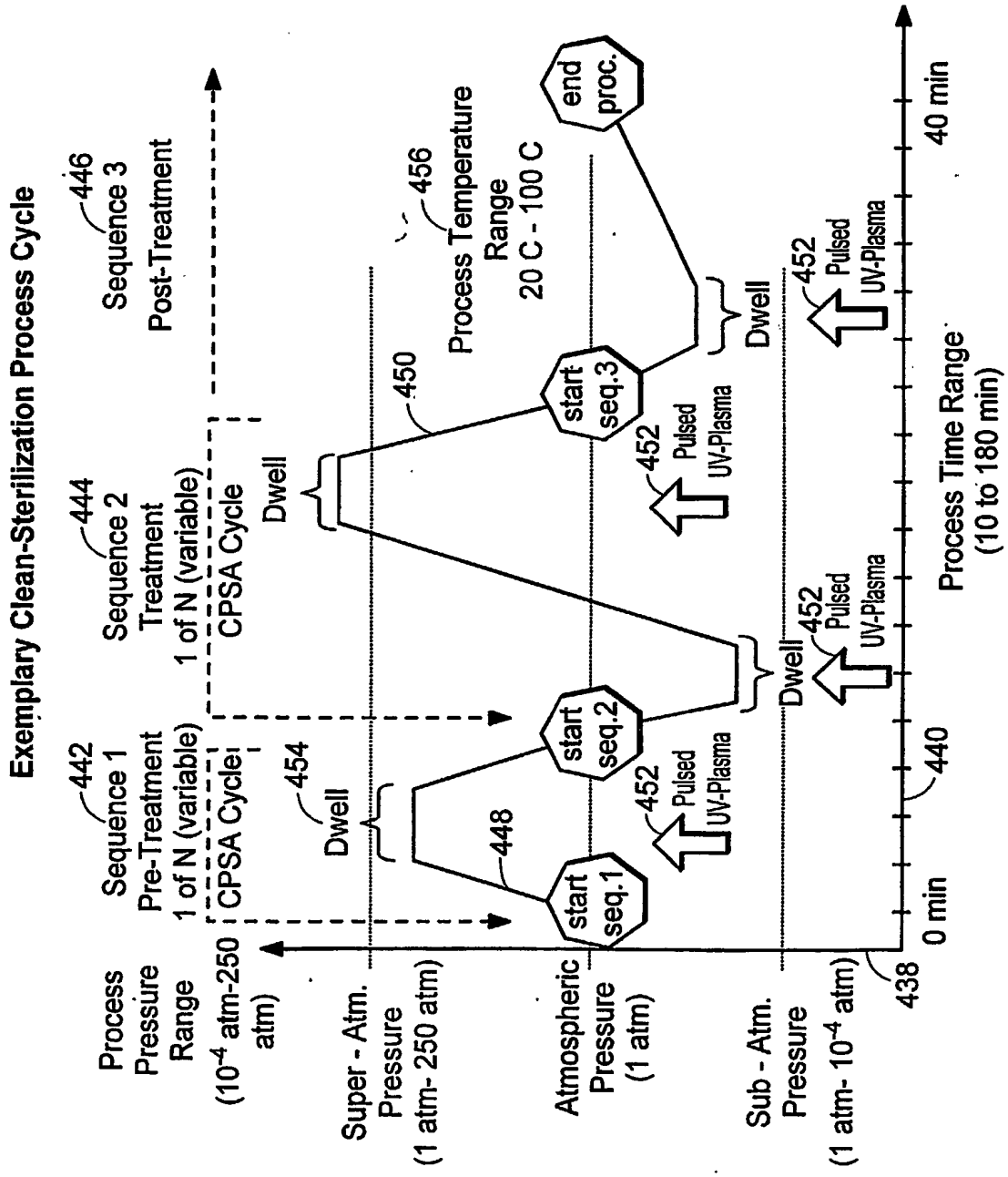

Exemplary Clean-Sterilization Process Cycle
Process Pressure Range (10^{-4} atm-250 atm)
Super - Atm. Pressure (1 atm- 250 atm)
Atmospheric Pressure (1 atm)
Sub - Atm. Pressure (1 atm- 10^{-4} atm)
442 Sequence 1 Pre-Treatment 1 of N (variable) CPSA Cycle
444 Sequence 2 Treatment 1 of N (variable) CPSA Cycle
446 Sequence 3 Post-Treatment
454 Dwell
448
450
456 Process Temperature Range 20 C - 100 C
start seq.1
start seq.2
start seq.3
end proc.
452 Pulsed UV-Plasma
Dwell
438
0 min
440
Process Time Range (10 to 180 min)
40 min

FIG. 24

Exemplary Clean-Sterilization Method (Aqueous PCA Wash)

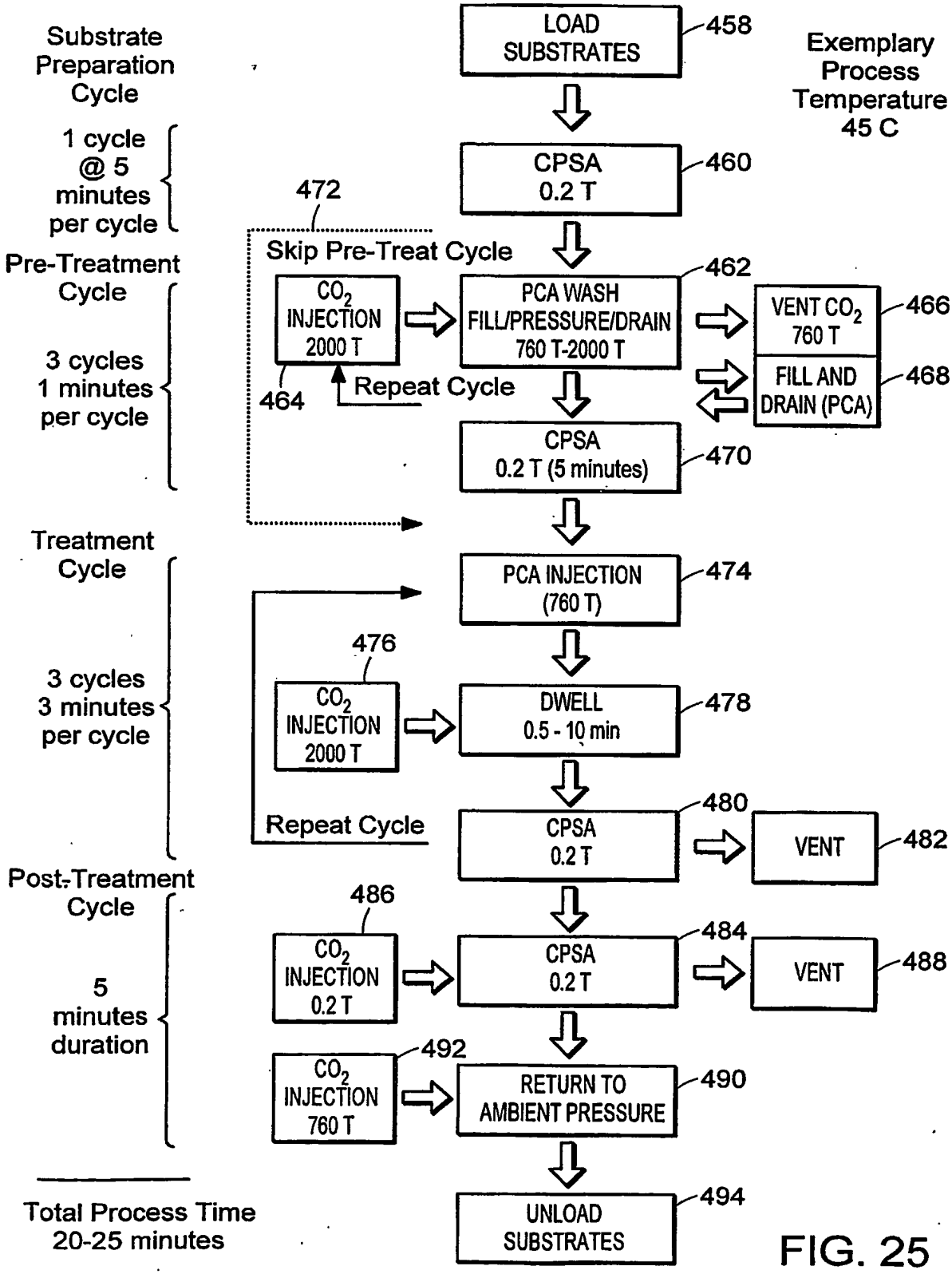


FIG. 25

Exemplary Clean-Sterilization Method (Dense Phase CO_2 Extraction)

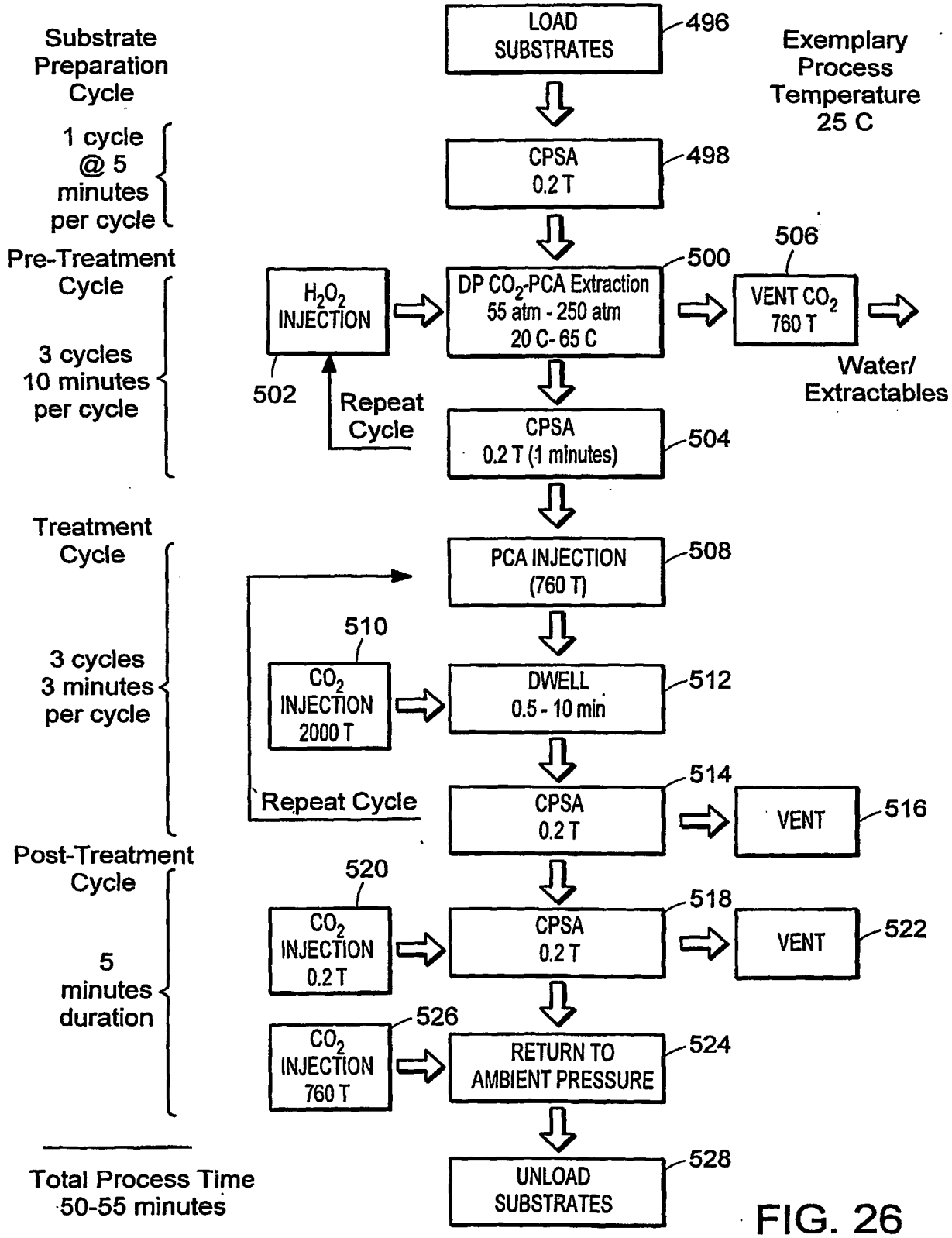


FIG. 26

Exemplary Clean-Sterilization Coating Method (Dense Phase CO_2 Extraction)

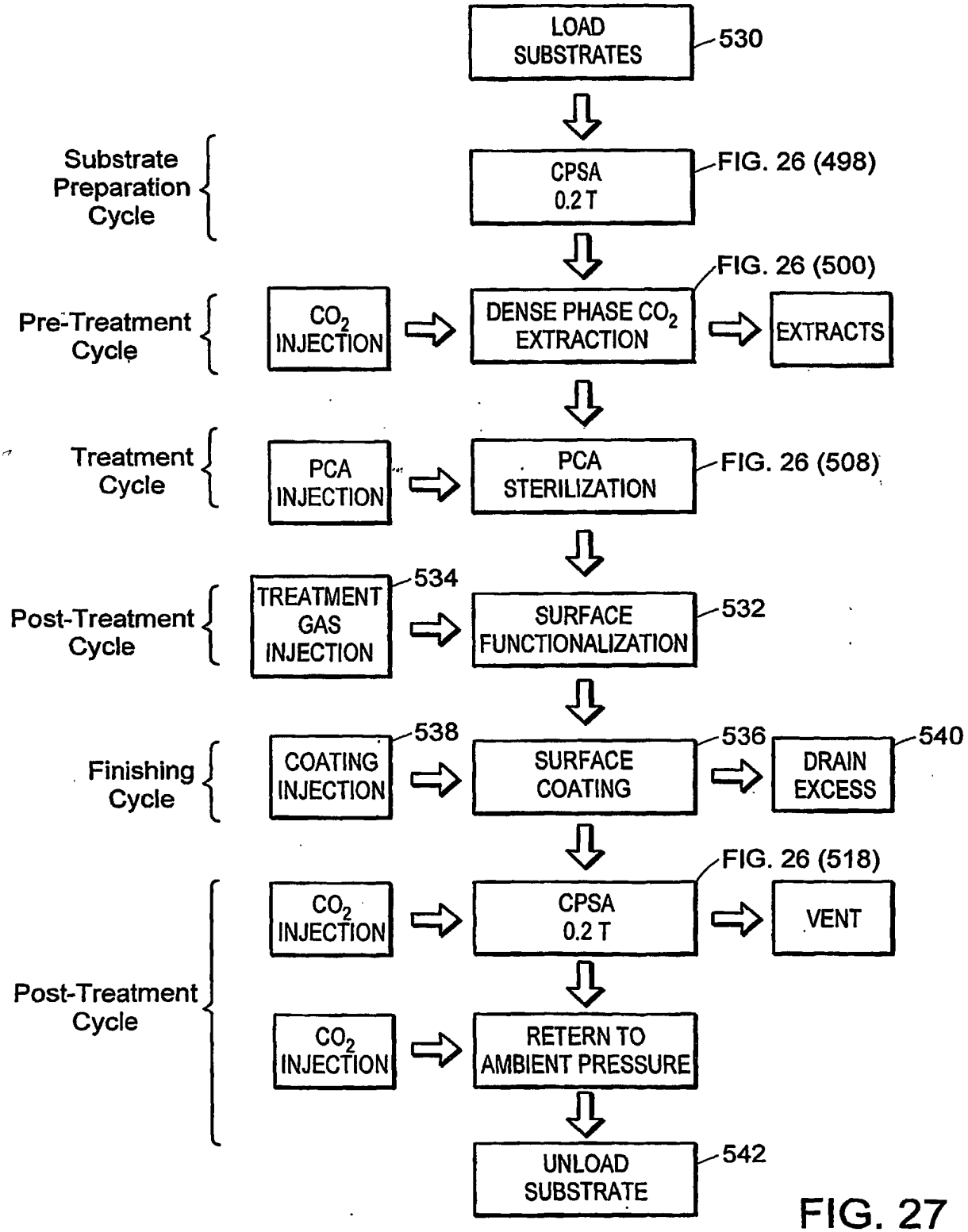


FIG. 27

Exemplary
Dense Fluid
Extraction Profile

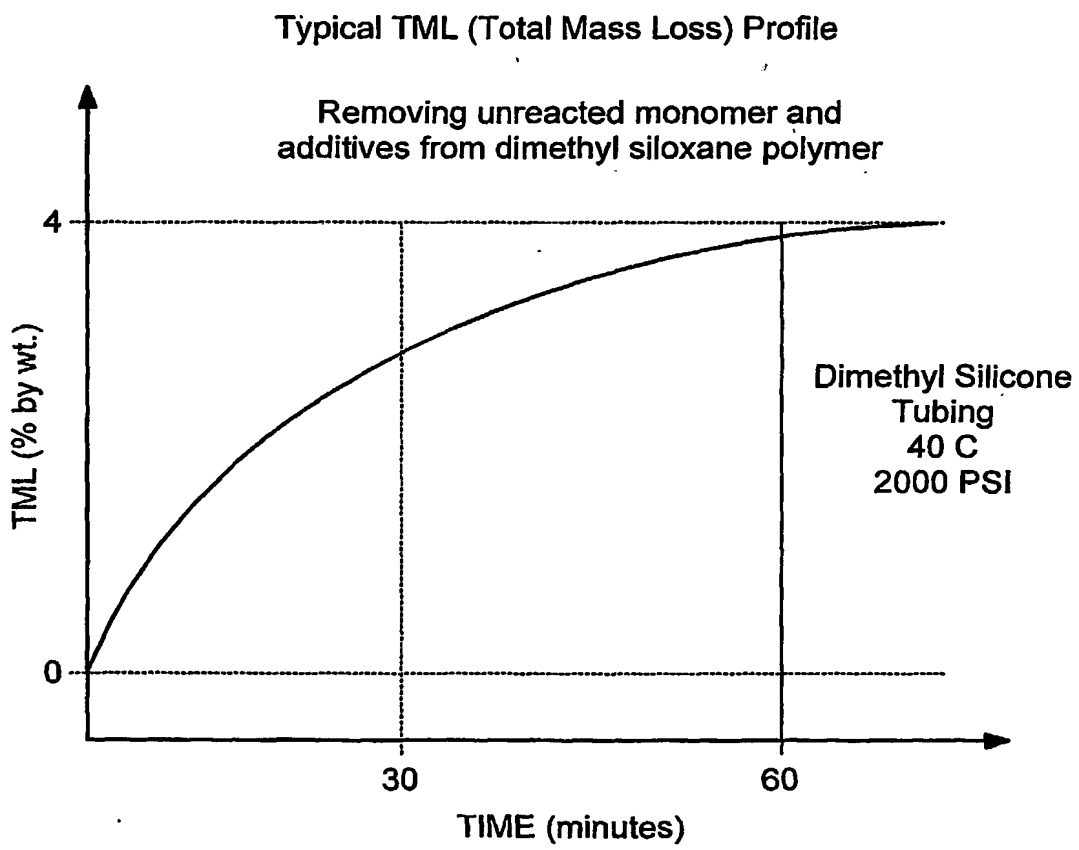


FIG. 28

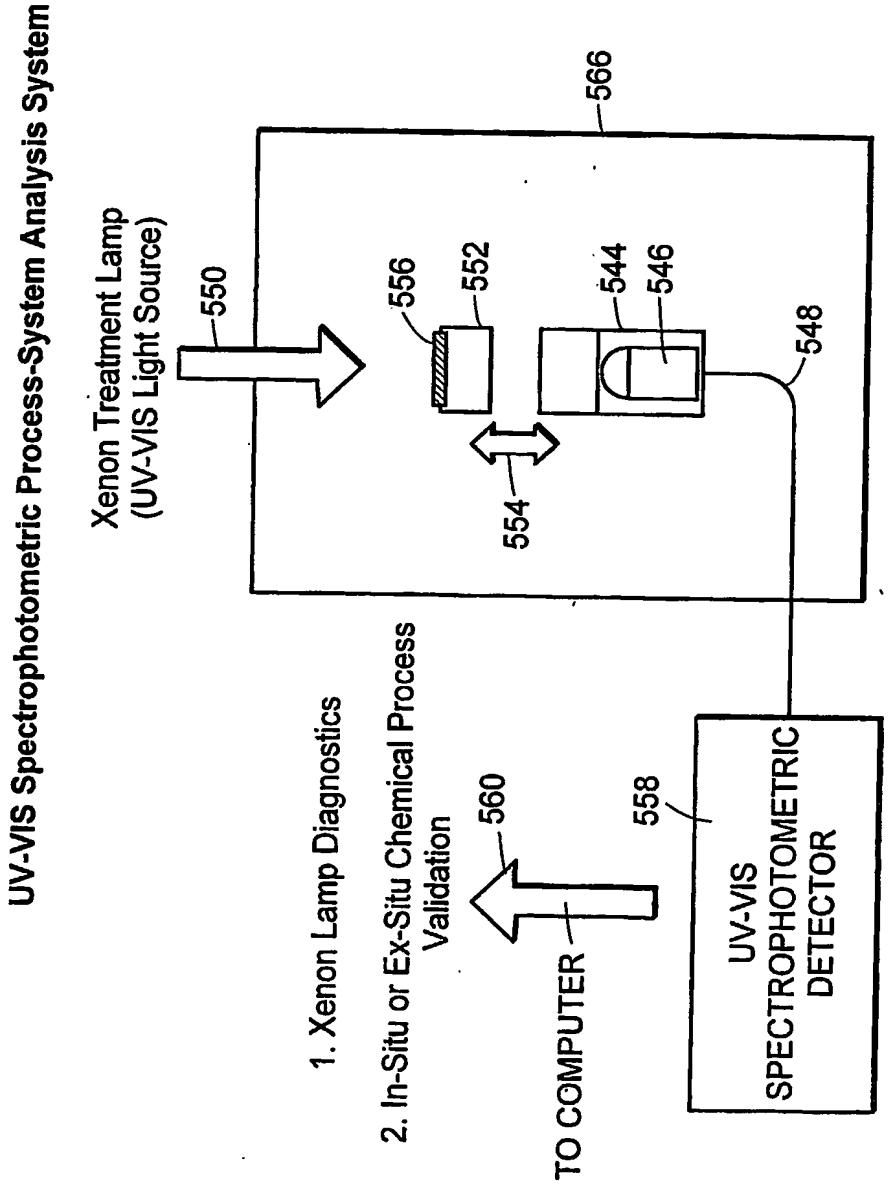

UV-VIS Spectrophotometric Process-System Analysis System
Xenon Treatment Lamp
(UV-VIS Light Source)
550
566
556
552
544
546
548
554
1. Xenon Lamp Diagnostics
2. In-Situ or Ex-Situ Chemical Process Validation
560
558
TO COMPUTER
UV-VIS
SPECTROPHOTOMETRIC
DETECTOR

FIG. 29A

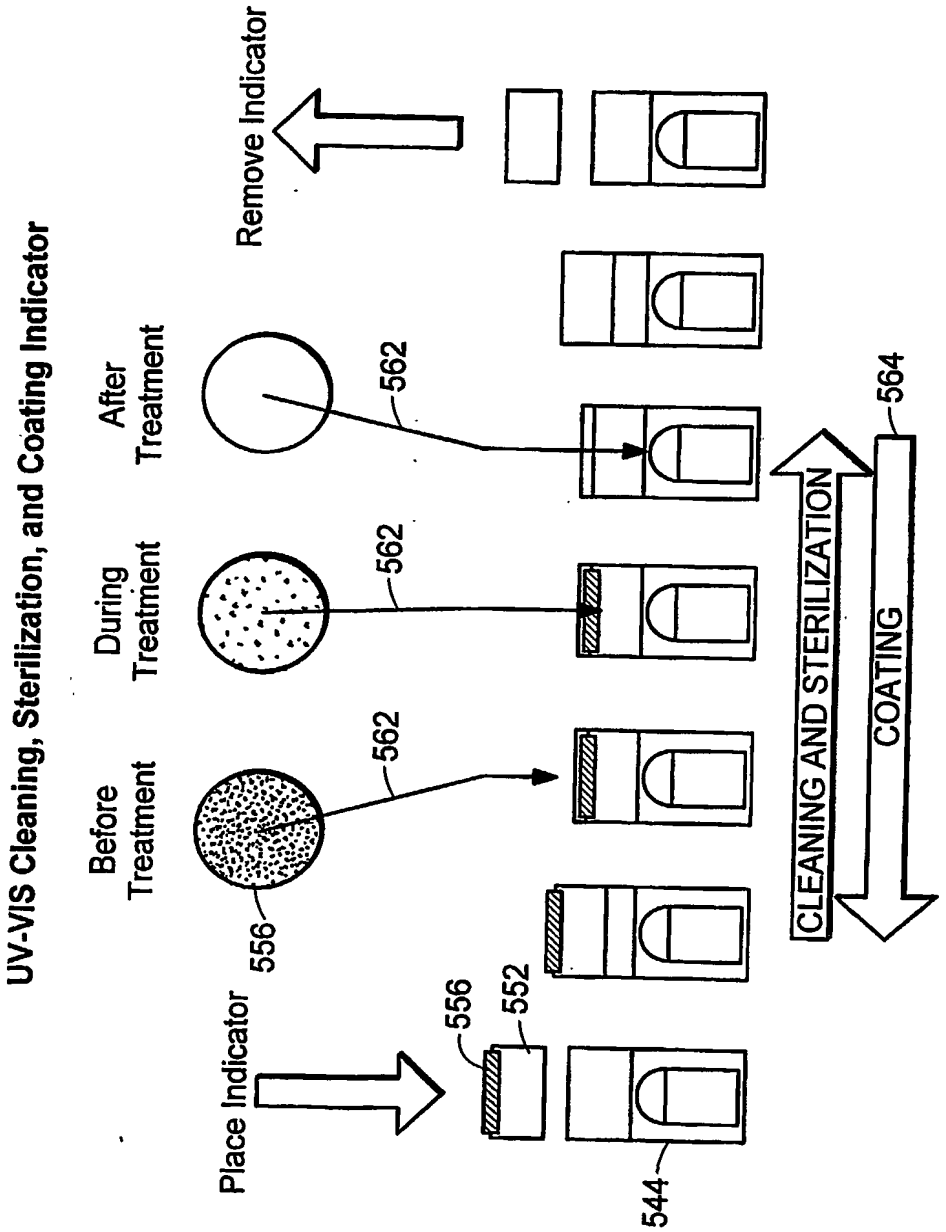

UV-VIS Cleaning, Sterilization, and Coating Indicator
Before Treatment
During Treatment
After Treatment
Remove Indicator
Place Indicator
556
562
552
544
564
CLEANING AND STERILIZATION
COATING

FIG. 29B

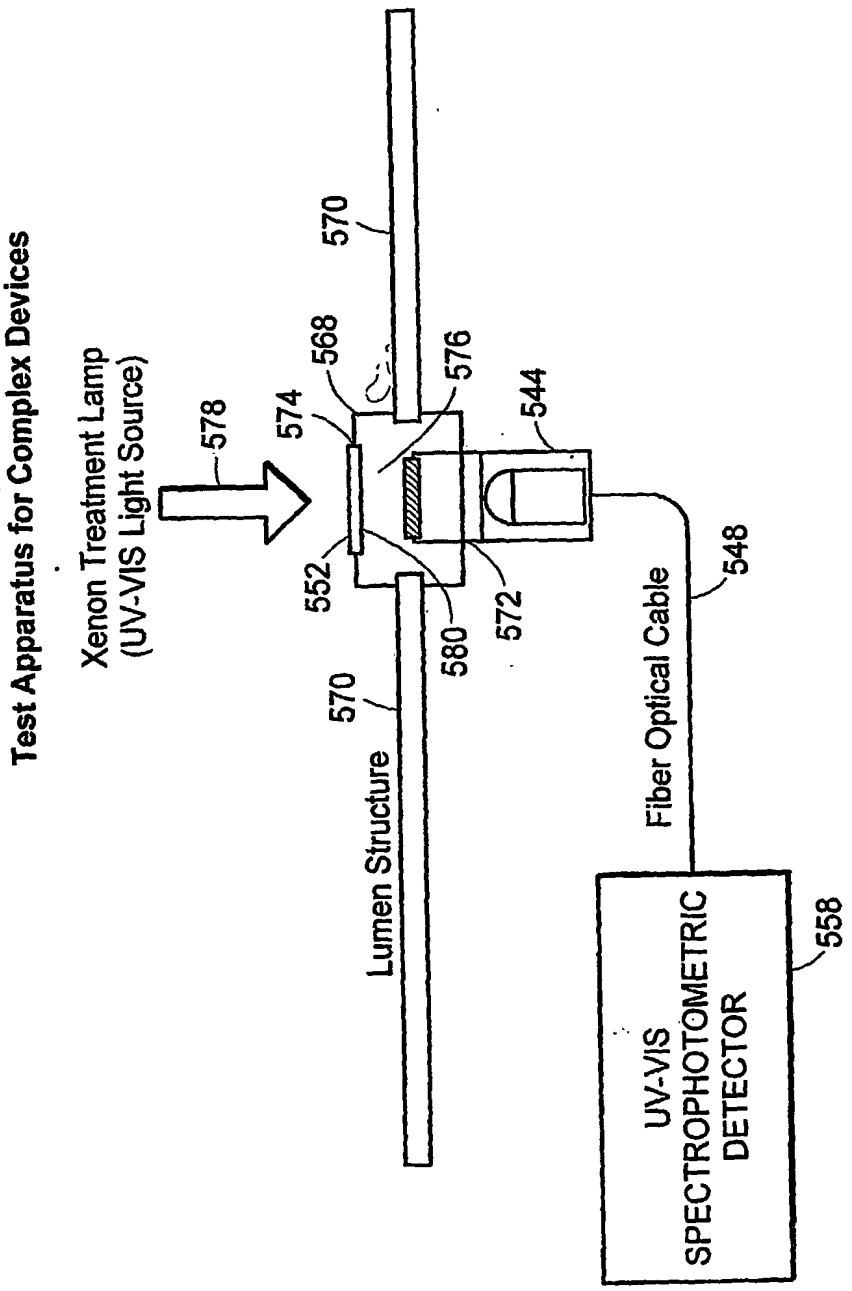

Test Apparatus for Complex Devices
Xenon Treatment Lamp
(UV-VIS Light Source)
578
574
568
570
576
544
552
580
572
570
548
Lumen Structure
Fiber Optical Cable
UV-VIS
SPECTROPHOTOMETRIC
DETECTOR
558

FIG. 30